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The Significance of

Metallic Constituents

in Textile Fabrics

By A. H. PETTINGER

THAT traces of iron and copper in textile fabrics are undesirable from the point of view of bleaching and dyeing, is a well-known fact. Thus the presence of small quantities of iron acting catalytically in a peroxide bleach liquor brings about irregularities and local over-oxidation, with the result that tendering of the goods frequently follows. Wool containing much iron is never stoved by sulfur dioxide satisfactorily, and not only do stains often appear but when subsequently dyed, such goods color unevenly and fade unequally. Copper is also a metal which every bleacher fears on account of its powerful catalytic behavior in the bleaching of textile fibers. At the same time, because of its ability to cause stains on wool, the scourer and dyer of woolen and worsted must be on his guard. The same two metals were also shown recently by Davidson (J. Tex. Inst., 1932) to be predisposing factors to tendering of cotton goods by caustic soda in the kier boil when air is also present.

The average technologist would have enough cares and troubles to worry about if iron and copper were the only deleterious metallic impurities occurring in textile goods, but according to the work of Risch (Textilb., 1934) chromium oxide is also a catalytically active substance in the hypochlorite bleaching of cotton, and so he must add still another name to the black list. Rags and recovered waste frequently contain wool which had been chromed and even the chroming of rayon and cotton as an after-treatment for logwood blacks and vat colors is not unknown. (Bedford, Brit. Pat. No. 263,222, has protected the after-chroming of acetate and viscose rayon.)

Manganese is fortunately not very commonly found in textile materials, but is none the less dangerous. As a catalyst it may cause losses and tendering in bleaching while its intense oxygen-carrying properties make even small traces undesirable in goods to be proofed with rubber or latex. (Mention may be made of mordanting cellulose acetate by manganese. French Pat. No. 570,264 and its use in bleaching cotton, E. P. 13,902.)

It is difficult to estimate how many different elements are made use of in the modern processes of dyeing, printing and finishing, but it is considerable. Titanium salts have for instance been much used in recent years for delustering viscose and acetate rayon; we have yet to discover whether their influence as chemical reagents in the other more usual textile processes are good or bad. (As regards bleaching, however, titanium compounds are known to have a stabilizing effect.) Barium compounds and oxides of tin are examples of other less common materials which have found use in textile processes recently, particularly delustering. A certain amount of difficulty is almost inevitably bound to arise should any waste goods containing these substances be made up into "new" fabrics or doctored and redyed. As an example of a process where rayon rags are worked up into textile fibers may be mentioned Brit. Pat. No. 359,012. The rags are first disinfected with peroxide and then treated with soda solution and pressed. They are then subjected to sodium acetate, later disintegrated in the usual way and then worked up as required. It would be a difficult matter to forecast the dyeing properties of a fabric manufactured in this way, containing silks from a variety of sources each delustered by some different process.

Tin oxide and tin salts have found employment in many modern processes, particularly for weighting natural silk and artificial rayons. Compounds of tin have also been applied to the delustering of fibers, for instance, the I. G. Farbenindustrie (Brit. Pat. No. 408,240, 1934) proposes to use sodium stannate in order to obtain on viscose a matt effect like cotton. Incidentally soluble stannates are stated to react towards peroxide bleach liquors as restrainers and stabilizers. The Roessler and Hasslacher Chemical Co., Niagara Falls, N. Y., have recently protected the use of sodium stannate in hydrogen peroxide bleaching for this purpose. It is hardly within the province of the present account to deal with the uses of tin as a catalyst in the polymerization and thickening of oils but

the possibility of the metal being present in fatty alcohol sulfonates and similar preparations might well give the thoughtful analyst a clue as to their mode of manufacture. There are instances in the literature where the presence of tin in textile goods is undesirable, however, and hence the analysis and examination of fabrics for the metal is occasionally called for.

Messrs. Goldman, Hubbard and Schoffschall in the U. S. Bureau of Standards Technical Paper No. 322, 1927, report that tin weighting accelerates the destructive action of sunlight on silk, particularly when that fabric had had contact with perspiration. Ohl in Monats Seide u. Kunstseide, 1933, finds that delustering treatment with sodium stannate and barium chloride brings about a slight increase of strength of rayon at first which falls away on storage. The ultimate result is a diminution of strength.

An Austrian patent was taken out as recently as 1927 which proposed the addition of metallic salts, e.g., lead acetate to reserve pastes in order to obtain colored reserve printings with vat dyes. As is well known, the interaction of lead salts with wool which has undergone treatment with strong alkalis tends to the formation of dark brown stains of lead sulfide. The application of lead salts would thus have to exclude all fabrics which contained animal fibers or which would come into contact with such material in later processes.

The proposal to use salts of zirconium, a rare metal in technical processes, is worthy of note (Ger. Pat. No. 422,097).

Curious compounds have been proposed from time to time in connection with moth-proofing wool and animal fibers. A claim made in 1926 relates to the use of insoluble rare-earth soaps, metals such as cerium, lanthanum and thorium being suggested. Strontium salts have been suggested for the same purpose (Brit. Pat. No. 365,233, 1932).

Two rather unusual patents of the Imperial Chemical Industries refer to the use of metallic nickel as a catalyst for the reduction of indigo to the leuco state by hydrogen. Readers will no doubt recall the fact that nickel is employed by the hydrogenation process or reduction of fats for rendering unsaturated and unstable oils less liable to oxidize, thicken or turn rancid.

The desire to be able to identify goods made by one's own firm at any future date has prompted the suggestion that metal salts of nickel, cobalt and others should be deliberately applied to textile fabrics in order to facilitate the recognition of manufactured goods if at any time dispute should arise as to their origin.

NOTES ON THE DETECTION OF METALS IN TEXTILES

The number of published papers on the examination of textiles for metallic radicles is large but unfortunately there is very little consistency between them, and the analyst who is new to the work is offered an embarrassingly large selection of methods to choose from. Many observers propose to ignite the fabric and test the residue; others prefer destruction with strong mineral acids, while still a third favor extraction of the sample a number of times with acids. Some sort of correlation between these processes is obviously called for and the inference is that some discrimination must be made between:

(a) Mineral matter occurring in a fiber naturally. For example, wool and animal fibers contain some quantity of lime and iron.

(b) Mineral impurities present from manufacture but non-removable by extraction without decomposition.

(c) Mineral matter acquired in the wet processes and weaving of the fiber. This impurity is, as a rule, merely adsorbed and not in chemical union with the fiber substance.

As an example of the latter class mention must be made of iron stains such as arise when the material comes into contact with damp metal or solutions of iron. Such contamination is simply dealt with and usually yields to acid treatment. Between the two extremes (a) and (c), (a) incapable of removable and (c) readily so, we have the indeterminate stage (b) where the foreign substance is united to the fiber in a fairly intimate way. This may be brought about by its chemical insolubility or through some chemical forces. Levi (Ann. Chem. analyst, 1927) has described the determination of copper in hemp, linen and cotton. Fibers which had received impregnation with copper soaps yielded the metal to extraction with nitric acid (classification c) while goods which had also been subjected to aniline black treatment and development with chrome retained their copper when treated thus (classification b), because the copper became converted into an insoluble form with the chromium. This example demonstrates the complexity of the subject.

Then let us consider wool or other animal fiber. Destruction of the organic matter by burning leaves an ash which contains all the mineral constituents of the original substance. Iron and lime are invariably found (classification a) and if a sample of wool suspected of contamination with iron were examined in this way it would be impossible to say whether the metal in the residue was derived from outside intervention as would be the case if the wool had undergone rust staining, or whether it could all be accounted for by the natural mineral content of the fiber. It is clear that as regards fibers such as are derived from animal sources and contain metals naturally, chemical examination must proceed along the lines of an *extraction*. In the case of wool, loose-adhering and foreign iron deposits may be removed by treatment with hydrofluoric acid for 30 minutes at 45° C., 5 to 10 per cent of this acid on the weight of the fabric in as concentrated a solution as possible can be relied upon to remove iron oxides or its salts without carrying away the minerals chemically combined as part of the fiber. In the case of artificial silk and fibers of the regenerated cellulose class, no such difficulty and confusion arises. The mineral content of viscose rayon is frequently as low as

2 mgm. per 100 gm. and so any appreciable quantity of residue or ash on burning can be ascribed to contamination arising from the processing.*

It will be seen from the above remarks that the question of how a contaminating metallic compound is isolated from a fabric is as important to the textile chemist as the subsequent detection and estimation of the elements themselves. While many analysts have studied the question of how copper, iron and such metals may be tested for and quantitatively determined, very little work has been done as yet to find in what manner those substances are united to the fiber. On the looseness or otherwise of their assoication with the chemical micelle or ultimate fiber unit must depend their influence on dyes, bleach liquors and so forth.

Perhaps when we have a better understanding of the relation between metals and textiles we shall be able to interpret certain apparently contradicting and conflicting results which appear in the literature of dyeing and bleaching on the subject of mineral contamination, and which have provoked lively discussion in times past.

*This, of course, does not apply to viscose rayon which has to be delustered by the introduction of metallic oxides.

The Shrinkability of Fabrics*

By KATHERINE FISHER†

IT was only yesterday, in the history of the manufacture of fabrics, that many women believed they could solve the problem of fugitive colors in wash fabrics only by "setting" these colors at home. The question was, what would best serve this purpose? Soaking the fabric in a salt or vinegar solution was a widely used method for "setting" color but many wrote us asking for a more effective method of making the color fast, as the salt and vinegar had failed to do the trick.

Less than ten years ago, surveys among our readers revealed the fact that there were those who firmly believed that no colored fabric could be made fast to sun. Others reported that colors sold as "fast" often faded in use, but without any exception, all who answered our questions were willing to pay more for colors that remained fast to tubbing and sun. Such colored fabrics are, of course, now generally available but it is quite obvious that the shopper needs a guide to such hidden qualities in the form of dependable identifying labels. Many have said to us that such a label would be invaluable to them.

Shrinkage of wash fabrics was and still is another vexing problem. Some months ago, we made a survey among our readers on this question. We found that some, indeed many, considered shrinkage a necessary evil. They were meeting the problem unsatisfactorily by "allowing for shrinkage" in various ways; by buying ready-made garments a size too large, washing the fabric before making it up or making garments "roomy to start with." Not a few thought that the soap used in laundering was to blame for shinkage or that the commercial laundry was at fault. Salespeople often told them this when they complained of excessive shrinkage in garments they had purchased. To our question, "Would you be willing to pay more for fabrics satisfactorily pre-shrunk by the manufacturer?" all but one of the hundreds answering us said "Yes" very emphatically. A typical answer to this question was "Oh, dear, yes; it would save us actual cash as well as our dispositions." Our survey gave us a vivid picture of the overwhelming preference for fabrics pre-shrunk satisfactorily by the manufacturer, but many questions were raised as to the dependability of any claims made for such pre-shrinking. It was quite apparent that a label in association with the manufacturer's trade-mark was considered more valuable to them than a salesperson's word. In fact, thoughtful shoppers apparently did not expect salespeople to take the responsibility of vouching for such service qualities in the absence of the manufacturer's identifying label.

Of course, neither the soap nor the laundry can prevent shrinkage in fabrics, except in the case of wool. Nor can the fading of fugitive colors be prevented in laundering fabrics either at home or at the commercial laundry. Both should be taken care of in the manufacture and finish of the fabric. And colored fabrics that have been pre-shrunk satisfactorily should also be fast-colored. The absence of one quality greatly discounts the value of the other.

It is obvious that fabrics to be pre-shrunk satisfactorily should not shrink out of size or fit after repeated launderings. In this respect we need uniform standards for shrinkage, as well as for fastness of color. We know that fabrics may be satisfactorily shrunk in their manufacture, during the finishing process. The sanforizing process for shrinkage is able to do this work effectively and is now being used in pre-shrinking a wide variety of cottons and linens. It is, of course, most important to the consumer that all fabrics pre-shrunk by such a controlled process should be clearly labeled.

In a recent survey we made among 2,500 women, as to their experiences with silk and rayon dresses, about one-third reported a shrinkage in dry cleaning, while at least half reported shrinkage when the washable types of silk and rayon dresses were washed. Without any exception, the answer was emphatically "Yes" to our question, "Would you like to know definitely what fabrics and qualities you are buying in dresses?" You and I know that there are on the market today silk and rayon fabrics of excellent quality and dresses made with these fabrics that can either be washed successfully or dry cleaned without shrinking out of size. It would be an invaluable

*Paper presented at 24th Annual Convention, National Retail Dry Goods Association, January 18, 1935.

†Director, Good Housekeeping Institute, New York City.

service to consumers to have these clearly identified for them. Some are so identified but many more are not. Such identifying labels would be of great sales value to you, and their wider use would undoubtedy raise standards in textile fabrics in general.

For years, shoppers have been able to buy certain types of fabrics and textile products carrying the brand names of manufacturers who, through the years, have steadily built up a reputation for making products of serviceable quality. There are, however, many fabrics, dresses, and other textile products which carry no brand name or no identification whatever as to serviceability.

I believe that you and I agree that shoppers need much better guides to hidden qualities in fabrics than they now have. That they would heartily welcome these guides we have proved in our various surveys. I am of the opinion that the time is not far off when this will be more available in the form of dependable labels giving information as to such service qualities as shrinkability, permanent finish, fastness of color, washability and cleanability. For you, these labels would have a tremendous sales value.

● S. O. C. M. A. MEETING

The regular monthly luncheon-meeting of the Synthetic Organic Chemical Manufacturers' Association was held on May 2nd at the Chemists' Club, New York City. The secretary's report concerned itself with reciprocal tariffs, inflation and the national industrial recovery act. It was also reported that reservations had already started to come in for the June outing at Skytop and that Lewis W. Douglas would be the guest speaker at the get-together dinner on that occasion.

● NATIONAL DYE

National Aniline & Chemical Co. has released a sample card on the following new dyestuff. Copies are available upon request.

National Diazine Fast Yellow 2G—an addition to the National line of developed dyes and said to produce with National Developer Z clear yellows of good fastness to washing and excellent fastness to sea water, perspiration, cross dyeing, steaming, alkali and rubbing. It is especially recommended by the manufacturers for discharge styles because of its excellent dischargeability on cotton and rayon. It is also said to be well suited for embroidery yarns and sewing threads, while its fastness to cross dyeing renders it useful for cotton, rayon and silk effect threads.

● SOLVAY PUBLICATIONS

Solvay Sales Corp. announces publication of the following booklets, copies of which are available upon request.

The Anchor Travels South—an attractive booklet describing the new plant of this company located in Baton Rouge, Louisiana. A George Annand map in colors showing the location of the plants of the Solvay Process Company in the United States is a feature of this booklet.

Solvay Liquid Chlorine—this booklet was reviewed on page 174 of our April 8th issue.

● WYANDOTTE

J. B. Ford Co. announces publication of the 10th edition of the following booklet. Copies are available upon request.

Wyandotte in the Textile Mill — a 72 page booklet describing some of the uses of Wyandotte Textile Sodas and Alkali and suggesting methods or formulas for application. It is divided into four parts as follows: Part 1—Wool, Part 2—Silk, Part 3—Cotton and Part 4—Miscellaneous. Included in Part 4 are several "textile suggestions" and "textile calculations." There are numerous illustrations.

● FADE-OMETER

Atlas Electric Devices Co. announces publication of a booklet on their latest model Fade-Ometer, the type FDA. Among the claims mentioned in the booklet are the following: faithfully duplicates and accelerates sunlight fading, permits testing of various size specimens—in steps or in stages, permits control of temperature and humidity and a 24-hour shut-off time clock enables the operator to set the Fade-Ometer to automatically shut off at any predetermined time which compensates for power interruptions and failure. Also included is a table of Fade-Ometer-sunlight equivalents for various seasons of the year and a list of users in various industries. Copies of this bulletin are available upon request.

● OBTAIN RIGHTS TO AQUA-SEC

Exclusive rights for the Aqua-Sec processing of silks and rayons for France, England, Italy and Switzerland have been granted to Gillet et Fils of Lyon, France, it was announced recently by Donald M. Martin, president of the Aqua-Sec Corporation.

Gillet et Fils, with their subsidiary companies, are said to be the largest dyers and finishers of silks and rayons in France, England, Italy and Switzerland. The subsidiary in England is Joshua Wardle, Ltd., Leek Staffs, England. The European dyers and finishers will process fabrics by the Aqua-Sec method exactly as is done in this country at the present time by the United Piece Dye Works.

● STAINLESS STEEL

Republic Steel Corp. has issued a folder dealing with the use of Enduro Stainless Steel in the textile industry. An interesting feature of this circular is a series of actual color illustrations of yarn samples dyed with acid colors and acid chrome colors showing how the stainless steel equipment holds the true color of the dye. Copies of this bulletin may be obtained from this journal.

PROCEEDINGS OF THE
American Association of Textile Chemists and Colorists

JUNE 3, 1935

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CALENDAR OF COMING EVENTS

Outing, Philadelphia Section, Torresdale Country Club, Torresdale, Philadelphia, June 7, 1935

* * *

Outing, New York Section, North Jersey Country Club, Paterson, N. J., June 28, 1935

Analytical Methods for a Textile Laboratory

The Council of the A.A.T.C.C., believing that Dr. Scott's series of articles entitled "Analytical Methods for a Textile Laboratory" might well be brought together in book form, have proceeded with its publication. Copies may be obtained from the Secretary of the Association, Harold C. Chapin, Lowell Textile Institute, Lowell, Mass., for 75 cents.

Proceedings of the American Association of Textile Chemists and Colorists

Fading of Dyeings in Radiation of Different Intensities[1, *]

By WM. D. APPEL

I. INTRODUCTION

OVER twenty years ago, Harrison[2] showed that radiation of high intensity does not have the same proportional effect on the colors of dyed fabrics as radiation of low intensity. He concluded that any attempt to shorten the time of exposure in laboratory tests for fastness to light[3], by increasing the intensity of the radiation beyond that at which dyeings will fade faster than they fade in sunlight, will give results of no practical value.

Nevertheless, the hope persists that a greatly accelerated light exposure test can be devised. For example, in 1928 the statement was made that "The ideal fading apparatus is one in which the light which produces fading is similar to sunlight but of greater and known intensity"[4]. In 1933, an attempt was being made to speed up the fading process many times by using a 200,000 candlepower arc with 1 hour of arc exposure equal to 15 hours of midday sunlight[5].

If the fading of dyeings exposed to radiation of high intensities where the fading is more rapid than in sunlight does not indicate the fastness to be expected in sunlight, the question arises: does the fading in sunlight correctly represent the fastness to be expected in relatively low intensity radiation, for example, diffused daylight, where fading is slower. Put in a different way, how can the results of exposures in the "standard sun test" of this Association or the results of the commonly used Fade-Ometer (glass-enclosed carbon arc lamp) tests be interpreted in terms of the use of materials in radiation of lower intensities, for example, in the diffused light in rooms?

The studies made several years ago by the Subcommittee on Light Fastness of the Research Committee of this Association[6] furnished information bearing on this question. They showed that 6.7 days of exposure in what was essentially a north window resulted in the same fading, *on the average,* as 10 hours exposure in the standard sun test, i. e. "at an angle of 45° from the horizontal, facing due south, between 9 a.m. and 3 p.m. on sunny days only, in a cabinet covered with a good grade of window glass approximately one-eighth of an inch thick, and open at the sides to allow free access of air to the samples"[7]. However, this ratio of 6.7 days to 10 hours is approximately correct for only 57 per cent of the 1252 dyeings tested and very large errors result when it is applied to many of the dyeings. For example, a 1 per cent dyeing on cotton of Primuline developed with Beta Naphthol required only 1 day of north window exposure to produce the fading obtained in 10 hours standard sun exposure; but a 2 per cent dyeing on cotton of Diazo Brilliant Green 3G developed with developer Z, required 17 days of north window exposure to produce the fading obtained in 10 hours standard sun exposure.

These differences in behavior may be attributable to the difference in intensity of the radiation (the illumination produced by the radiation is, on the average, of the order of 8100 foot-candles in the standard sun exposure and 1200 foot-candles in the north window exposure). They may also be attributable to differences in the quality of the radiation, temperature, atmospheric humidity, or a combination of these.

The present work was undertaken to find out what part the intensity of the radiation plays in the fading of a few selected dyeings when the conditions of exposure are under control. The effect of a change in atmospheric humidity on the fading of the dyeings was also studied.

This work is a part of the program of the Subcommittee on Light Fastness of which Wm. H. Cady is Chairman. The experiments were carried out at the National Bureau of Standards. Wm. C. Smith, Research Associate for the Association started the work. It was continued by Warren E. Emley, Jr. and completed by Robert J. Hunn. The whole-hearted devotion of these men to the rather tedious task of obtaining and reducing the data is gratefully acknowledged.

II. PROCEDURE

The seven dyeings used in this study were chosen because of the behavior of similar dyeings in previous light exposure tests[6]. These previous dyeings faded, when exposed continuously in what was in effect a north window, either decidedly more or decidedly less than was to be expected, the basis of judgment being the fading of

* Presented at the Annual Meeting, December 8, 1934. Publication approved by the Director of the National Bureau of Standards, U. S. Department of Commerce.

these dyeings relative to the fading of many others when all were exposed simultaneously in the standard sun test and simultaneously (but for a different period of time) in the north window test. The seven dyeings are listed and the behavior of the previous dyeings when exposed in the north window and also in the Fade-Ometer are indicated in table I.

The present dyeings were exposed in an insulated box having a Vita glass window facing the glass-enclosed carbon-arc of the Fade-Ometer[8]. The temperature of the air in the box was maintained at approximately 43° C by a thermostat and the relative humidity was maintained at approximately 75 per cent with a saturated solution of sodium chloride[9]. Some tests were made at 31 per cent relative humidity obtained with a saturated solution of magnesium chloride[9]. The box was placed so that the dyeing faced the arc at a distance from it which would make the intensity of the radiation at the sample equal to that in the Fade-Ometer, or to 0.33, 0.1, or 0.02 times this intensity[10].

The spectral reflectance of each dyeing at each of 7 wave lengths in the visible spectrum was measured before exposure and at intervals of time during the exposure, the samples being removed from the box for measurement and returned to it to continue the exposure[10]. The wave lengths were 405, 436, 501, 546, 578, 651, and 703 millimicrons. The method of measuring reflectance is described elsewhere[12].

TABLE I. Dyeings Studied and Summary of Results Obtained

Colour Index No.[13]	Serial Number of dyeing in series 4.[8]	Dyeing	Fading obtained in exposure indicated below compared with fading obtained in standard sun exposure for equivalent time. (Results from Series 4)[9]. Fade-Ometer	North Window	Wave length selected for comparison shown in next column Millimicrons	Ratio of hours of exposure at intensity indicated to hours at intensity 1.0 to produce approx. equal change at the given wave length Intensity 0.3	0.1	0.02	Ratio of hours of exposure at 31% relative humidity to hours at 75% rel. humidity to produce approx. equal change at the given wave length
735	...	2% Erio Green Extra, Dyed acid, wool	....	....	651	..	8.5	..	1
735	442	1% Erio Green Extra, Dyed acid, wool	more	less					
...	...	0.5% Erio Chrome Cyanine RC Top-chromed wool	....	....	578	..	10.	..	1
...	804	Ditto	equal	more					
922	...	2% Methylene Blue 2B, soap and acetic acid silk	....	....	651	3	5.5	..	1
922	239	Ditto (dyed with soap)	equal	less					
...	...	2% Diazo Brilliant Green 3G, developer Z, cotton	....	....	651	3	9.	..	1
...	24	Ditto	slightly less	less					
662	...	1% Brilliant Green Cryst Tannin, Cotton	....	....	651	1	3.5	13	1.6
662	992	Ditto	equal	more					
768	...	2% Eosine Y, acetic acid silk	....	....	546	1	2.	50	1.9
768	864	Ditto	less	more					
812	...	1% Primuline NAC, developer Beta Naphthol, cotton	....	....	501	1	1.5	11	2
812	11	Ditto	slightly less	more					

III. RESULTS AND DISCUSSION

The results are shown graphically in the figures. The curves in the upper part of each figure represent the spectral reflectance of the dyeings before exposure and after exposure for the periods of time indicated. The observed values for the spectral reflectance at one wave length lying in the absorption band of each dyeing are plotted in the lower part of each figure against time of exposure to obtain curves showing the change in reflectance at that wave length. These curves may be considered to indicate the relative rates of fading under the different conditions of exposure, though, as explained later, they may be only a rough indication when the fading under the different conditions did not take the same course.

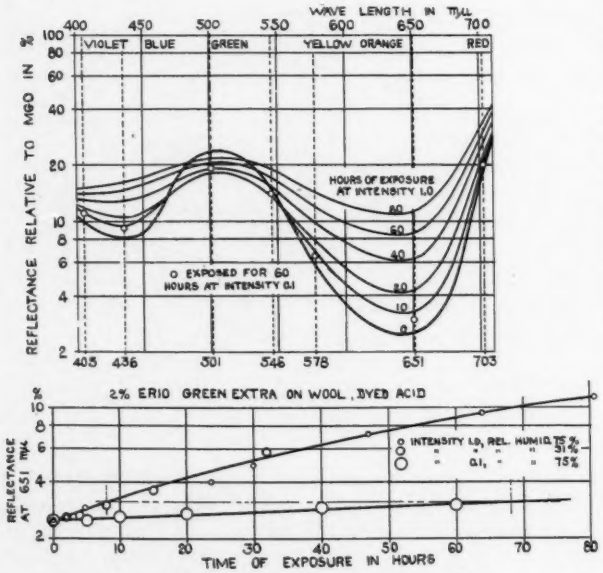
WAVE LENGTH IN mμ
400 450 500 550 600 650 700
VIOLET BLUE GREEN YELLOW ORANGE RED
REFLECTANCE RELATIVE TO MGO IN %
HOURS OF EXPOSURE AT INTENSITY 1.0
80
60
40
20
0 EXPOSED FOR 60 HOURS AT INTENSITY 0.1
405 436 501 546 578 651 703
2% ERIO GREEN EXTRA ON WOOL, DYED ACID
REFLECTANCE AT 651 mμ
INTENSITY 1.0, REL. HUMID. 75 %
" " " 31 %
" 0.1, " 75 %
TIME OF EXPOSURE IN HOURS

Fig. 1—2% Erio Green Extra, dyed acid on wool.

The interpretation of figure 1 will be discussed at some length for the benefit of those who are not familiar with spectral reflectance curves.

The upper part of this figure represents the fading of a 2 per cent dyeing of Erio Green Extra on wool when it is exposed to the radiation of the glass-enclosed carbon arc in a position at which the intensity of the radiation was designated arbitrarily as intensity 1 (Fade-Ometer intensity) in an atmosphere of 75 per cent relative humidity. Before exposure the dyeing reflects about 25 per cent as much green and blue-green light as the standard white magnesium oxide surface. It reflects less than 10 per cent as much violet and less than 4 per cent as much orange. Thus the dyeing is bluish green in color.

Upon exposure for 10 hours, the reflectance increases in the violet and in the yellow to red regions of the spectrum as a result of the destruction of some of the dye under the photochemical action of the radiation. It *decreases* in the green region. The net result is that the color of the dyeing becomes duller. Further exposure results in an increase in reflectance throughout the visible spectrum and the color becomes lighter and lighter.

When the dyeing is exposed at a greater distance from the arc, where the intensity of the radiation is 0.1 of that previously used, it fades in the same way but at a slower rate. The reflectance after 60 hours exposure at intensity 0.1 is indicated in the upper part of figure 1 by the small circles. These circles lie between the curves for 0 and 10 hours exposure at intensity 1.0 and near the latter. Thus, the dyeing has faded somewhat less in 60 hours exposure at intensity 0.1 than in 10 hours exposure at intensity 1.0.

The curves in the lower part of figure 1 indicate how the reflectance at wave length 651 millimicrons, where the dye on the fiber is absorbing the most light, changes as the time of exposure increases. The increase in reflectance for 8 hours exposure at intensity 1.0, upper curve, is seen to be about the same as that for 60 hours exposure at intensity 0.1, lower curve. Evidently, under the conditions of the experiment, more of the radiant energy incident upon the dyeing at intensity 0.1 is utilized in producing fading than that incident upon the dyeing at intensity 1.0. That the 2 per cent dyeing on wool of Erio Green Extra fades at the same rate in air of relative humidity 75 per cent and 31 per cent (intensity 1) is shown in the lower graph in figure 1.

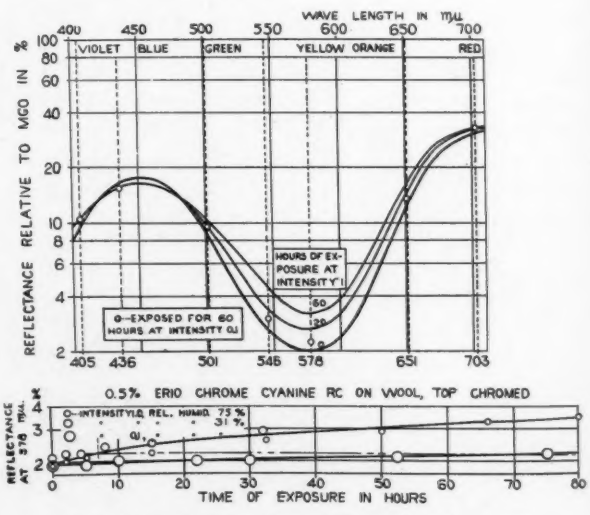
WAVE LENGTH IN mμ
400 450 500 550 600 650 700
VIOLET BLUE GREEN YELLOW ORANGE RED
REFLECTANCE RELATIVE TO MGO IN %
HOURS OF EXPOSURE AT INTENSITY 1.0
0—EXPOSED FOR 60 HOURS AT INTENSITY 0.1
405 436 501 546 578 651 703
0.5% ERIO CHROME CYANINE RC ON WOOL, TOP CHROMED
REFLECTANCE AT 578 mμ
INTENSITY 1.0, REL. HUMID. 75 %
" " " 31 %
" 0.1, " 75 %
TIME OF EXPOSURE IN HOURS

Fig. 2—0.5% Erio Chrome Cyanine RC, top-chromed, on wool.

The results for a 0.5 per cent dyeing on wool of Erio Chrome Cyanine RC, top chromed, given in figure 2, show that an exposure at intensity 0.1 which is 10 times as long as that at intensity 1.0 is required to produce equal change in reflectance at wave length 578 millimicrons.

There appears to be little or no humidity effect on the fading of this dyeing from 31 to 75 per cent relative humidity.

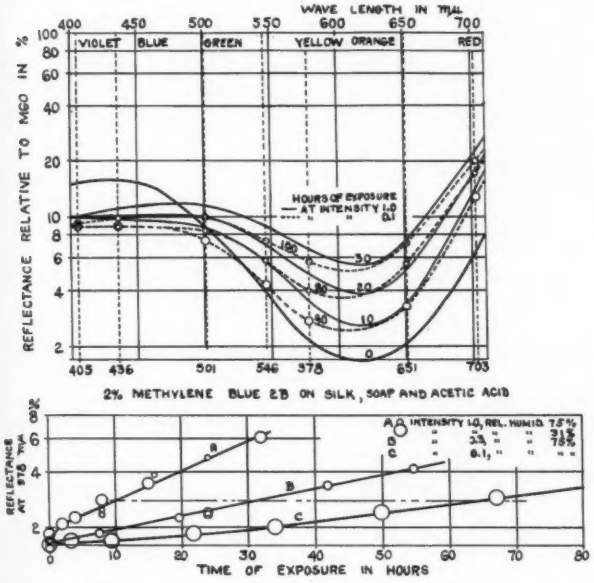
WAVE LENGTH IN mμ
VIOLET
BLUE
GREEN
YELLOW ORANGE
RED
REFLECTANCE RELATIVE TO MgO IN %
HOURS OF EXPOSURE AT INTENSITY 1.0
" " " 0.1
2% METHYLENE BLUE 2B ON SILK, SOAP AND ACETIC ACID
REFLECTANCE AT 578 mμ
A INTENSITY 1.0, REL. HUMID. 75%
B " " 31%
C " 0.1, " 75%
TIME OF EXPOSURE IN HOURS

Fig. 3—2% Methylene Blue 2B, dyed with soap and acetic acid, on silk.

The character of the fading of a 2 per cent dyeing on silk of Methylene Blue 2B, dyed with soap and brightened with acetic acid, is quite different in exposures at intensities 1.0 and 0.1. This is shown in the upper part of figure 3 by the difference in the solid and dashed curves. The change in reflectance in 100 hours of exposure at intensity 0.1 approximates that obtained in 20 hours exposure at intensity 1.0 for wave lengths 405, 436, 501, and 546 millimicrons and approximates that obtained in 30 hours exposure at intensity 1.0 for wave lengths 651 and 703. Because of this behavior and the fact that "fading", or change in color as seen by the eye, takes into consideration the behavior at all wave lengths in the visible spectrum, a comparison of reflectances at neither wave length 651 millimicrons nor 546 millimicrons gives a true picture of the fading as seen by the eye. The change in reflectance at wave length 578 millimicrons is intermediate and was used in the lower part of figure 3 as a rough indication of the effect of intensity. At this wave length, the exposure at intensity 0.1 requires about 6.5 times the time of an exposure at intensity 1.0 to produce the same change in spectral reflectance. However, at intensity 0.33, three times the time of exposure at intensity 1.0 is required. There is no difference in rate of fading of this dyeing in atmospheres of relative humidity 75 per cent and 31 per cent, judged by the reflectance at wave length 578 millimicrons.

The results obtained with a 2 per cent dyeing on cotton of Diazo Brilliant Green 3G, developed with developer Z, require no special comment. Exposures at intensities 0.33 and 0.1 respectively of approximately 3 and 9 times that at intensity 1 are required to produce equal fading. The same change in spectral reflectance at wave length 651 millimicrons was obtained in equal exposures at 75 per cent and 31 per cent relative humidity.

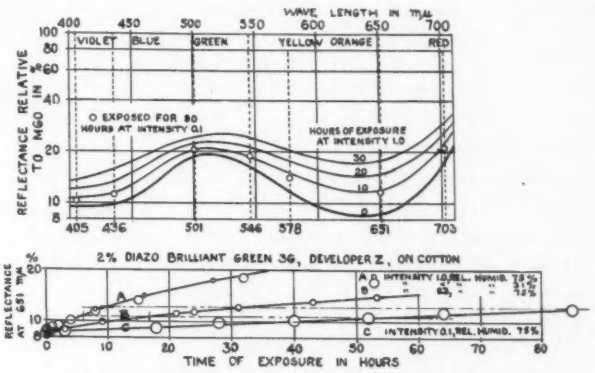
WAVE LENGTH IN mμ
VIOLET
BLUE
GREEN
YELLOW ORANGE
RED
REFLECTANCE RELATIVE TO MgO IN %
O EXPOSED FOR 80 HOURS AT INTENSITY 0.1
HOURS OF EXPOSURE AT INTENSITY 1.0
2% DIAZO BRILLIANT GREEN 3G, DEVELOPER Z, ON COTTON
REFLECTANCE AT 651 mμ
A INTENSITY 1.0, REL. HUMID. 75%
B " " 31%
C INTENSITY 0.1, REL. HUMID. 75%
TIME OF EXPOSURE IN HOURS

Fig. 4—2% Diazo Brilliant Green 3G, developer Z, on cotton.

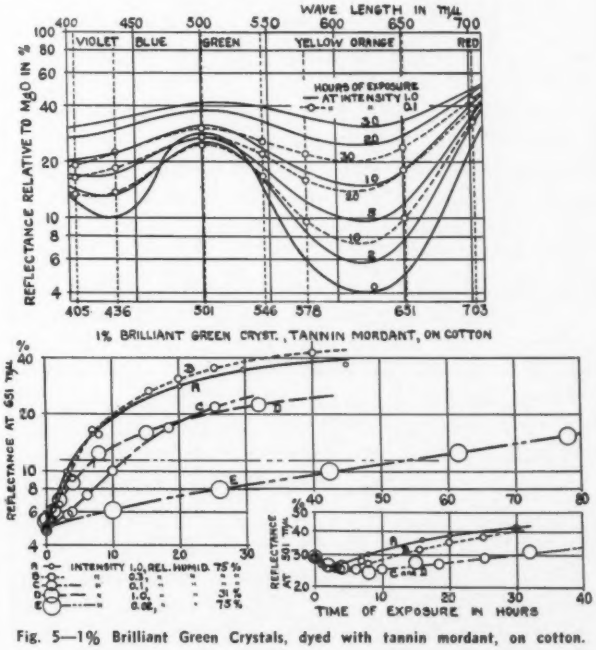

Fig. 5—1% Brilliant Green Crystals, dyed with tannin mordant, on cotton.

The behavior of a 1 per cent dyeing on tannin mordanted cotton of Brilliant Green Crystals, shown in figure 5, is in sharp contrast to that of the dyeing of Diazo Brilliant Green, just discussed. Approximately the same change in the spectral reflectance of this dyeing at wave length 651 millimicrons in a given time of exposure is obtained at intensity 1 and intensity 0.33. An exposure at intensity 0.1 of 3.5 times that at intensity 1 was required to produce equal change in reflectance at wave length 651 millimi-

crons. An exposure at intensity 0.02 of only 13 times that at intensity 1 was required to give the same result.

Decreasing the relative humidity of the atmosphere from 75 to 31 per cent increased the time required to produce equal change in reflectance at wave length 651 millimicrons by a factor of about 1.6.

The change in reflectance with time of exposure for the 1 per cent dyeing on cotton of Brilliant Green Crystals at wave length 501 millimicrons is shown in figure 5 in order to show how the reflectance decreases and then increases at this wave length and for comparison with the curves for reflectance at wave length 651 millimicrons.

The differences in behavior of this dyeing at different wave lengths under the different conditions of intensity and humidity limit the conclusions to be drawn from the behavior at any one wave length.

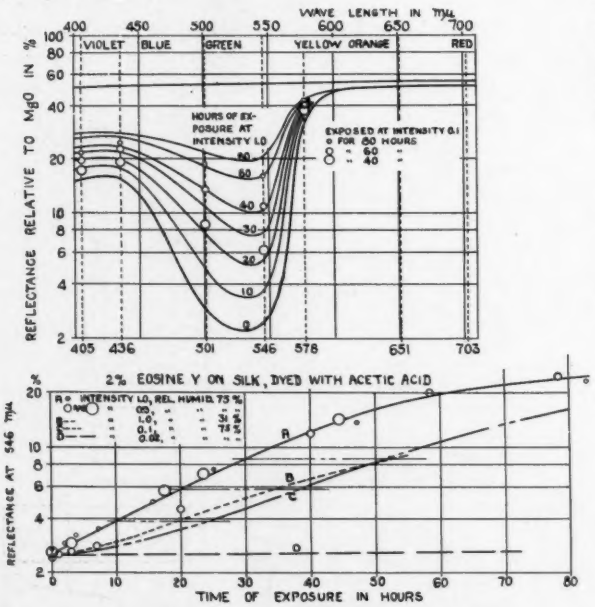

Fig. 6—2% Eosine Y, dyed with acetic acid, on silk.

The results for a 2 per cent dyeing on silk of Eosine Y dyed with acetic acid are shown in figure 6. This dyeing behaves somewhat like the dyeing of Brilliant Green Crystals. Evidently, the same change in reflectance at wave length 546 millimicrons with time of exposure was obtained at intensity 1.0 and intensity 0.33. Instead of taking 10 times as long at intensity 0.1 as at intensity 1.0 to produce a given change in reflectance at 546 millimicrons, only 2 times as long was required. However, when the intensity was reduced to 0.02, the time required was about 50 times that at intensity 1.0. The dyeing faded nearly twice as fast at relative humidity 75 per cent as at relative humidity 31 per cent.

A 1 per cent dyeing on cotton of Primuline developed with Beta Naphthol judged by its reflectance at wave length 501 millimicrons, behaved similarly to the dyeing of Eosine on silk at wave length 546 millimicrons except that at intensity 0.02 the first required 11 times the exposure given at intensity 1. to produce a given change in reflectance.

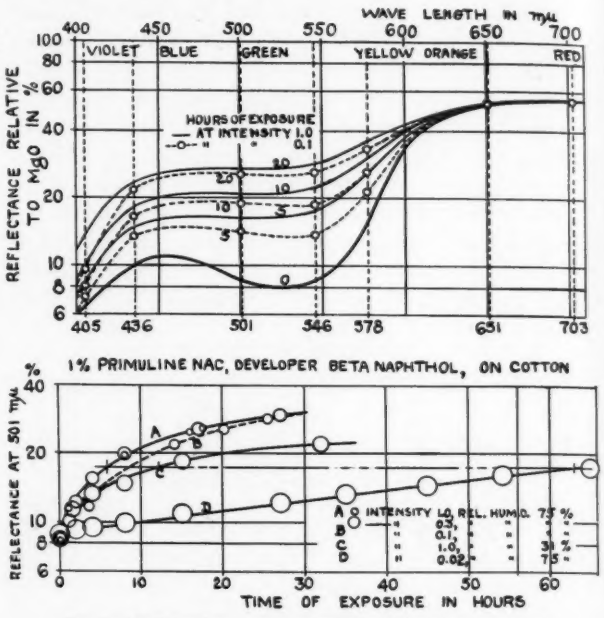

Fig. 7—1% Primuline, developed with Beta Naphthol, on cotton.

The results for all of the dyeings are summarized for ready comparison in table I. It is seen that decreasing the intensity of the radiation from 1 to 0.33 may increase the time of exposure required to produce the same change in spectral reflectance at a given wave length three times. On the other hand it may not increase the time of exposure required at all. Thus, if the dyeings of Diazo Brilliant Green 3G and Eosine Y were exposed simultaneously at intensity 1, the relative fastness would be found to be quite different from the relative fastness judged by similar exposures at intensity 0.33.

Decreasing the intensity of the radiation from 1 to 0.1 may increase the time of exposure required to produce the same change in spectral reflectance at a given wave length 10 times or only 1.5 times. Decreasing the intensity to 0.02 may increase the time 50 times or only 11 times.

Four of the dyeings studied fade just as fast in air of relative humidity 31 per cent as 75 per cent. However, one dyeing takes twice as long at 31 per cent as at 75 per cent to produce the same change in spectral reflectance at the selected wave length.

The dyeings of Primuline, Eosine Y, and Brilliant Green, faded to a given extent in a shorter time at intensity 0.1 than was to be expected. It is of interest to note in Table I that these same dyeings faded more than was

to be expected in the north window test, i.e., in a condition of low average light intensity. All three of these dyeings fade faster when the relative humidity of the air is higher, as it was in the north window test. Perhaps the reason why the dyeing of Methylene Blue behaved like that of Diazo Brilliant Green in the north window test rather than like those just discussed is because its fading is not accelerated by an increase in humidity from 31 per cent to 75 per cent. There is reason to believe that the fading of the dyeing of Erio Chrome Cyanine is accelerated by humidities above that studied. If so, this might explain the fact that it faded more than was to be expected in the north window test.

IV. CONCLUSIONS

The results obtained lead to the conclusions given in the following statements:

1. The change in reflectance of some dyeings on exposure to light is not proportional to the intensity of the incident radiation, other conditions being unchanged.

2. The time of exposure required to produce a given amount of fading at intensity 0.1 may be anywhere from 10 times to only 2 times that required at intensity 1. This finding is based upon exposures to the radiation of the glass-enclosed carbon arc, with unit intensity defined as that used in the Fade-Ometer. (An exposure of 1 hour in the Fade-Ometer produces the same fading of dyeings, on the average, as an exposure of 1.3 hours in the standard sun test of the Association.)

3. The rate of fading of some dyeings is not affected by a change in the relative humidity of the surrounding atmosphere of from 75 per cent to 31 per cent but the rate of fading of others may be retarded by a factor of 2.

4. The relative fastness to light of dyeings when exposed under one set of conditions; intensity of the radiation, humidity, etc., is not necessarily their relative fastness when exposed under some other set of conditions. This fact should be remembered when the results of comparative exposures in the standard sun test, Fade-Ometer, etc., are being interpreted

NOTES AND REFERENCES

[1]The terms "radiation" and "intensity of radiation" are used in this paper in accordance with the definition for "intensity of radiation in transit" given in the International Critical Tables Vol. 1, p. 38 as follows: "The amount of radiant power per unit area which passes through an element of area which is normal to the direction of propagation; this equals the volume density of radiant energy at the point considered."

[2]W. Harrison. The action of light on coloring matters. J. Soc. Dyers and Colourists, *28*, 225-236 (1912).

[3]The terms "light," "sunlight," and "daylight" are used to include invisible as well as visible radiation.

[4]Anon. The fastness to light of dyed materials. Canadian Textile J., *45*, 667-669 (1928).

[5]Anon. The Shirley Institute fading station. Dyer, *70*, 535-536 (1933).

[6]Wm. H. Cady and W. D. Appel. Report of the Sub-committee on light fastness. 3. The fading of dyed textiles in daylight and in carbon-arc light. Am. Dyestuff Rept. *18*, 407-446 (1920).

[7]1933 Yearbook. Am. Assocn. Textile Chemists and Colorist. p. 116.

[8]Manufactured by the Atlas Electric Devices Company, Chicago, Illinois.

[9]Hugh M. Spencer. Laboratory Methods for Maintaining Constant Humidity. Int. Crit. Tables *1*, 67, (1926).

[10]The distances from the arc to the sample were calculated on the basis of the inverse square law and the assumption that the window transmitted 90 per cent of the radiation incident upon it.

[11]A better procedure would have been to use a fresh sample for each exposure but this would have required a much longer time and more material than was available.

[12]Wm. D. Appel. A Method for measuring the color of textiles. Am. Dyestuff Reptr., *17*, 49-54 (1928).

[13]Colour Index. Society of Dyers and Colourists. Jan., 1924.

DISCUSSION

President Rose. This very interesting paper merits quite considerable discussion. It seems to me it opens up a great many contentious questions and I should like to hear from anybody who wishes to ask Mr. Appel a question.

Mr. Karl M. Herstein: Mr. Appel, were these tests all made with the Fade-Ometer?

Mr. Appel: Not with the Fade-Ometer. We used the arc. The samples were not exposed in the Fade-Ometer as such. They were exposed in a box, where we had rigid control on temperature and humidity.

Mr. Herstein: The point I want to bring out is that you have no comparison between the spectral distribution of your light source and sunlight as regards these effects.

Mr. Appel: No; we were not comparing the source there. These were all made with the same source and we have reason to believe that in the experiments we made in sunlight we will have similar effects there.

Mr. Herstein: But you don't know if the ratio between different intensities for this light source would be the same as the ratios for various intensities of sunlight?

Mr. Appel: No.

Dr. Hunter: I should like to ask if you tie in with your experiments what effect these factors of time had on different depths of dyeing. I believe some English work has pointed out that differences come from that score alone.

Mr. Appel: The experiments of the Committee made earlier show a very great effect of the depth of dyeing, but this was a rather laborious job, making all these measurements, and we had to restrict it. We worked just with these seven dyeings and did not study different depths.

Dr. Hunter: Were these on deeply dyed samples, or just tinted ones?

Mr. Appel: They were medium.

Mr. G. S. Hiers: I should like to ask what you mean by "medium"—one per cent, or half a per cent?

Mr. Appel: Well, I should have pointed out what they were. I think I have a list of them here.

There was a 2 per cent dyeing of Erio Green Extra, dyed acid on wool; a 2 per cent dyeing of Methylene Blue 2B, on silk; a half per cent dyeing of Erio Chrome

(Continued on page (202) 314)

Proceedings of the American Association of Textile Chemists and Colorists

Selective Adsorption from Soap Solutions

By HARVEY A. NEVILLE* and MILTON HARRIS†

I. INTRODUCTION

THE existence of an acid soap resulting from the hydrolysis of soap in dilute aqueous solution has been suggested by different investigators. Its formation has been represented by Williams[1] as follows:

$$(NaSt) \rightleftarrows NaSt \rightleftarrows St^- + Na^+ \rightleftarrows Na^+ + (St^-)$$

neutral colloid (sodium stearate) | simple molecules | + H^+ + OH^- | + H_2O | ionic micelle

$$x\ NaSt.HSt \rightleftarrows + \quad - HSt \quad NaOH$$

acid soap

The acid soap is probably also ionized as a colloidal micelle.

The addition of alkali to the soap solution obviously represses this hydrolysis. It is also apparent that the hydrolytic equilibrium will be disturbed by contact with any material which preferentially adsorbs fatty acid, acid soap or alkali. There is considerable evidence[1] to show that the principal surface-active constituent of a soap solution, to which is attributed its efficiency as a lubricant, detergent and emulsifying agent, is the acid soap. This view is consistent with the fact that the addition of alkali to a neutral soap solution raises the surface tension and decreases the effectiveness of the soap in the processes mentioned, while the addition of fatty acid often improves the emulsifying and lubricating ability of the soap solution.

If the acid soap is the surface-active agent in the solution, it will concentrate at interfaces and the aqueous medium will become more alkaline in contact with chemically neutral or inert materials. However, when the material forming an interface with the soap solution is acidic in nature or strongly adsorbs OH^- ions, the aqueous solution may become more acidic since the selective adsorption of OH^- ions may exceed the opposing effect due to the removal of acid soap.

Soap is adsorbed from aqueous solution at all types of interfaces, but the ratio of fatty acid to alkali in the adsorbed portion varies with the nature of the interface.

Soap foam, continuously removed from an aqueous solution by CO_2-free air was found to be less alkaline than the original solution while the latter became progressively more alkaline. The addition of alkalies to the soap solution decreased the selective effect and modified the character of the foam.

When air, carbon black, paraffin oil and pure olive oil were brought in contact with soap solutions, the aqueous medium became more alkaline and acid soap was adsorbed at the interface.

Fuller's earth, olive oil containing free fatty acid, wool and silk in soap solutions adsorbed soap and alkali at the interface and the aqueous medium became more acid. Cotton showed no selective adsorption and adsorbed a relatively small amount of soap.

The results of these experiments are consistent with the theory of the existence of an acid soap, resulting from the hydrolysis of soap in dilute solution.

II. RESULTS AND DISCUSSION

The qualitative effects obtained with various types of interfaces in dilute soap solution are shown in Table I.

The soap used in all experiments was a neutral pure olive oil soap which showed equivalent amounts of alkali and fatty acid upon analysis. The soap solutions were prepared with freshly-boiled distilled water. The concentration was 0.25 per cent and give a pH value of 10.0.

Adsorption in Foam—Thin films of soap solution in contact with CO_2-free air were continuously formed and separated from a soap solution by the means illustrated in Figure 1. The stream of air was first dried by passage through granular calcium chloride and concentrated sulfuric acid, then passed through a tower of soda lime to remove carbon dioxide and finally rehumidified to avoid evaporation of the soap solution. Two liters of soap solution was placed in the first aspirator bottle and with this amount of solution, a foam-height of 20 cm. was required before the foam passed into the collecting bottle. The rate of the air stream was regulated to maintain a slow, steady delivery of foam, permitting good drainage of the films so that a "dry" foam was obtained. The delivery tube had an internal diameter of 8 millimeters and the foam extruded from this built up into a very stiff mass in the collecting bottle, and remained unbroken for several hours. When the delivery end of this tube was constricted to 1 millimeter in diameter, the foam broke to a spray in the collecting bottle and samples of the resulting liquid could be taken at once. Some results obtained by this method are shown in Table II.

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†Research Associate at the National Bureau of Standards from the American Association of Textile Chemists and Colorists.

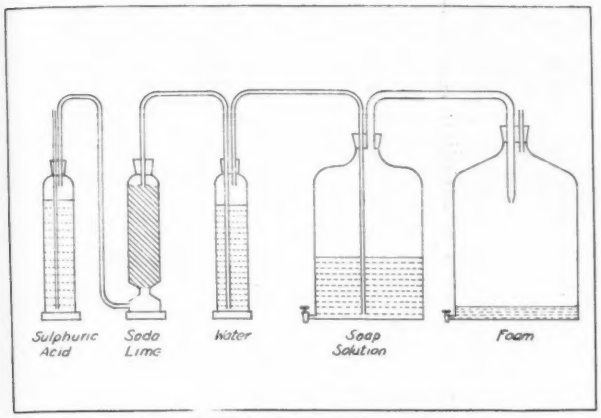

Fig. 1—Apparatus for preparation of foam.

TABLE I

Selective Adsorption at Various Types of Interfaces in 0.25 Per cent Soap Solution

Material in contact with soap solution:	*Aqueous medium becomes more:*	*Adsorbed at interface*
Air (foam)	alkaline	acid soap
Carbon (lampblack)	alkaline	acid soap
Fuller's earth	acid	soap and OH^-
Paraffin oil (medicinal)	alkaline	acid soap
Olive oil[a]	alkaline	acid soap
Olive oil[b]	acid	soap and OH^-
Wool	acid	soap and OH^-
Silk	acid	soap and OH^-
Cotton	no change	soap

[a] Contained 0.33% of free fatty acid.
[b] Contained 7.90% of free fatty acid.

TABLE II

The pH Values and the Soap and Sodium Carbonate Concentrations of Solutions and of the Foams Formed by Bubbling CO_2-free Air Through the Solutions

Description of Sample	*Concentration %*	*pH*
Original soap solution	0.25 soap	10.0
First 40 ml of broken foam	1.13 soap	9.6
Sample of 25 ml of broken foam after total of 250 ml removed	1.12 soap	9.6
Remainder of original solution	0.08 soap	10.5
Original solution of soap and soda ash	0.25 soap 0.50 soda ash	11.0
First 25 ml of broken foam	0.98 soap 0.45 soda ash	10.8

It is apparent from these data that the films of bubbles formed from a dilute soap solution contain a higher concentration of a solute which is less alkaline than the original soap, and the residual soap solution becomes progressively less concentrated and more alkaline as the foam is removed. In other words, the solute which is adsorbed at the air-solution interface is a product of the hydrolysis of soap which does not carry equivalent amounts of sodium hydroxide and fatty acid. The solution resulting from the broken foam exhibits a marked turbidity which clears upon the addition of alkali.

The direction of this selective adsorption is the opposite of that reported by Zacharias[2] who found that the residual soap solution became progressively more acid as successive increments of foam were produced and removed. The final solution had a pH value of 6.5 and would no longer lather. He apparently did not test the foam itself and mentioned no precautions against contamination by carbon dioxide.

It may be noted in Table II that the composition of the foam is approximately constant, changing very little in soap concentration and not measurably in pH value, while the original soap solution decreases to one-third its original strength and becomes more alkaline. This constancy of composition may be taken as evidence of a saturated film of the surface-active constituent of the soap solution.

When soda ash is added to the soap solution, the hydrolysis is repressed and a much less stable foam results. In this case a more rapid stream of air is required to raise the foam sufficiently to carry it over into the collecting bottle. There is less time for drainage and a larger quantity of solution is carried by each bubble. In spite of these disturbing factors, a selective adsorption in the same direction is observed and it is seen that while the soap is positively adsorbed the added alkali is negatively adsorbed.

Adsorption by Carbon—A sample of lampblack*, weighing 1.5 grams was stirred into 100 ml. of a 0.25 per cent soap solution. After standing for 30 minutes this suspension was filtered. The filtrate showed a pH value of 10.1. While this is not a large change, it was apparent that the carbon had removed some of the acid soap from the solution.

Adsorption by Fuller's Earth—When a sample of fuller's earth was mixed with distilled water (pH = 5.9) and filtered, the filtrate showed no change in hydrogen-ion concentration. When 5 g. of fuller's earth was stirred into 100 ml. of 0.25 per cent soap solution (pH = 10.0) and filtered, the filtrate had a pH value of 9.2. The selective adsorption of alkali by fuller's earth is well known, for when a solution of sodium chloride is filtered through it, sodium hydroxide formed by hydrolysis is removed and the filtrate becomes acid.

Adsorption by Oil Droplets—An emulsion was prepared from equal volumes of pure paraffin oil (medicinal) and the 0.25 per cent soap solution. This was permitted to stand in a separatory funnel until it had creamed but had not broken. The lower aqueous layer was drawn off and it was found to have increased in alkalinity to a pH value of 10.4.

This experiment was repeated using a pure olive oil in place of the paraffin oil. The olive oil was found to contain 0.33 per cent of free fatty acid. After creaming the aqueous layer had a pH value of 10.3. A commercial olive oil which contained 7.90 per cent of free fatty acid was emulsified in the same manner as the other oils. It formed a much more stable emulsion and the aqueous layer after

*Grade J. L. Martin Company, Philadelphia, Pa.

creaming had decreased in alkalinity to a pH value of 5.8. The above results indicate a selective adsorption of the acid products of the hydrolysis of soap or a negative adsorption of hydroxyl ions by oil droplets, except when the oil contains a considerable amount of free fatty acid.

Adsorption by Wool, Silk and Cotton—Two-gram specimens of purified* wool, silk and cotton were soaked in 100-ml. portions of the 0.25 per cent soap solution. At intervals of time, the pH value of each solution was determined with the glass electrode. The results are recorded in Table III.

TABLE III

pH Values of 0.25 Per cent Soap Solutions in Contact With Wool, Silk and Cotton for Various Lengths of Time

Time	pH of Soap Solutions		
	Wool	Silk	Cotton
0	10.0	10.0	10.00
1 min.	9.98	9.94	10.00
5 mins.	9.94	9.90	9.97
10 mins.	9.94	9.80	—
15 mins.	9.90	9.75	9.92
30 mins.	9.81	9.59	9.93
1 hour	9.66	9.36	—
17 hours	9.08	8.91	9.90

A similar set of experiments were made except that aliquots were taken from each solution at stated intervals, and analyzed for total alkali and fatty acid content. Only one aliquot was taken from each solution. The total alkali was determined by direct titration with standard acid, using methyl orange as the indicator. The total fatty acid was obtained by the procedure described by Levitt[5]. The values in Table IV are given in millimoles per gram of conditioned fiber and each is the average of three determinations.

TABLE IV

Adsorption of Alkali and Fatty Acid by Wool, Silk, and Cotton in 0.25 Per cent Soap Solution. The Values Given are Millimoles per Gram of Fiber Weighed in Equilibrium with an Atmosphere of 65% Relative Humidity at 70° F.

Time	Wool		Silk		Cotton	
	NaOH	Oleic Acid	NaOH	Oleic Acid	NaOH	Oleic Acid
	Millimoles		Millimoles		Millimoles	
5 mins.	.035	.009	.072	.066	.010	.013
15 mins.	.058	.014	.159	.079	.012	.014
30 mins.	.063	.018	.161	.084	.013	.011
1 hour	.073	.028	.165	.098	.012	.013
2 hours	.085	.032	.168	.103	.012	.012
24 hours	.107	.040	.178	.114	.015	.011

The results in Tables III and IV show that both wool and silk exhibit a pronounced selective adsorption of alkali, while cotton absorbs equal but relatively small quantities of both alkali and fatty acid. Silk adsorbs the greatest amount of alkali. This is to be expected since its low isoelectric point (pH 2.5) [3] shows it to be one of the most acid proteins known. It is of interest to note that the soap solutions became turbid almost immediately after coming in contact with the wool and silk, while the solution in contact with cotton remained practically clear for 24 hours.

*White, worsted yarn, prepared from raw wool which had been extracted with solvents and washed with water was used in this work. The yarn was extracted with alcohol and ether for six hours each, then washed with water and conditioned at 65 per cent relative humidity and 70° F. The preparation of the purified silk has been described elsewhere[3,4].

References

[1] R. C. Williams "Boundary Lubrication By Soap Solution." J. Phys. Chem. vol. 36, p. 3108 (1932).
[2] L. Zacharias. Jerome Alexander's "Colloid Chemistry," vol. 4, p. 656 (1932). Chemical Catalog Company.
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FADING OF DYEINGS

(Continued from page (199) 311)

Cyanine on wool top-chromed; 2 per cent Diazo Brilliant Green 3G, with developer Z, on cotton; 1 per cent Brilliant Green Crystals, tannin, on cotton; 2 per cent Eosine Y on silk; 1 per cent Primuline NAC, Betanaphthol, on cotton. That is the list of dyes.

Mr. Walter C. Durfee: I thought perhaps Mr. Appel had some interesting explanation of the discrepancies between theory and practice, some compound phenomena—

Mr. Appel: I don't think it interferes with theory at all.

Mr. Durfee: Is it due to a combination of light and chemical action at the same time?

Mr. Appel: Well, the fading of the dye presumably is chemical, induced by the action of the light, photo-chemical reaction.

President Rose: It seems to me that there is one point which comes from these observations that Mr. Appel is reporting to us and that is, his what we may call "aberrant" results belong in the range of colors that are frightfully poor to light, anyway.

Mr. Appel: I think that may be true (laughter) but, of course, we had to select dyeings where we could make a little progress. (Laughter.) We picked ones that would fade.

Mr. P. H. Stott: I wonder if the effect of dyeing Brilliant Green Crystals on silk would show upon the results obtained. I noticed that the test was made on tannin mordanted cotton.

Mr. Appel: Well, the Methylene Blue on silk had different characteristics from the Brilliant Green. They are not exactly related, but I think it would make a difference, yes.

Mr. Stott: I mean a comparison of the same dyestuff at different times.

Mr. Appel: In all probability it would make a decided difference.

Proceedings of the American Association of Textile Chemists and Colorists

Report of the Sub-Committee on

The Chemistry of Wool

By ALBAN EAVENSON, *Chairman*

THE accompanying research report of Milton Harris upon this subject indicates that, from the chemical viewpoint, wool is a very reactive substance. It is particularly susceptible to the action of alkalies and shows a marked preferential adsorption for alkali from solutions of alkaline substances. For example, from an ordinary soap solution wool removes about three times as much alkali as fatty acid. This fact shows the practical necessity of adding extra alkali continuously or at intervals to a wool scouring liquor to maintain the maximum efficiency of the soap.

The injurious effect of alkaline solutions is of wide practical importance in view of the processing treatments which wool undergoes in being converted from raw stock to the finished product, in addition to the alkaline laundering which wool fabrics may receive in use. For this reason many studies of the action of akallies upon wool have been made, but principally in terms of the resulting physical effects. Data obtained by Dr. Harris show that breaking-strength tests do not indicate alkali damage since the strength of wool yarn may actually increase as the result of alkali treatment, due to the matting of the fibers, although the yarn may be harshened and partially dissolved. The reflectance and compression resilience tests give more consistent measurements, but even these fail to detect damage until it has become all too apparent and serious.

It would appear that chemical action must be the fundamental basis of the damage to wool caused by alkalies, the physical effects becoming observable only when the chemical action has progressed to a sufficient degree. If this be the case, incipient damage must occur to a considerable extent before it becomes apparent or measurable by physical means. Thus it is highly desirable to develop methods to predict, detect and prevent serious damage before it

The accompanying Report of the Sub-committee on the Chemistry of Wool, presents an interpretative review of work done during 1934 by Milton Harris, a research associate of the A.A.T.C.C. working at the U. S. Bureau of Standards.

The personnel of the sub-committee is as follows:

Alban Eavenson, Chairman
Wm. D. Appel, Textile Section, U. S. Bureau of Standards
Werner von Bergen, Chemist, Forstmann Woolen Co.
G. E. Hopkins, Technical Director, Bigelow-Sanford Carpet Co.
H. A. Neville, Professor of Chemistry, Lehigh University
Donald H. Powers, Chemist, Rohm & Haas Co.
Robert E. Rose, Director, Technical Laboratory, E. I. du Pont de Nemours & Co.

This sub-committee has acted in advisory capacity and, under the active and able chairmanship of Alban Eavenson, has inaugurated a research program which, it is confidently expected, will aid in solving some of the complicated problems of textile processing, which apparently involve the chemistry of wool fiber to a greater extent than was heretofore believed.

LOUIS A. OLNEY,
Chairman, Research Committee.

thorough quantitative study of the nature of the action of the nature of the action of alkalies upon wool was undertaken. The committee is convinced that suitable methods of analysis and control of the degradation of wool by alkali can be based only upon primary chemical knowledge of the exact mechanism of this chemical reaction.

In the course of this investigation a surprising fact was discovered which seems to offer the most promising lead to the solution of this chemical problem. It was found that although the atomic ratio of nitrogen to sulfur in wool is about 10 to 1, these elements are removed from wool in the ratio of about 1 to 1 by the action of an alkali during the first stages of its attack. If wool protein were simply being dissolved, there should be ten atoms of nitrogen in the solution for every atom of sulfur, but the analyses showed that the sulfur was present in a much greater proportion. About 40 per cent of the sulfur content of the wool is lost during an alkaline treatment which dissolves only about 9 per cent of the wool, but thereafter the sulfur is removed in proportion to the wool dissolved. For example, treatment of wool yarn with a solution of caustic soda which is one-fourth normal (1 per cent by weight) reduces the sulfur content of the wool from its original value of 3.16 per cent to 1.85 per cent in 20 minutes at 150° C. The same result is obtained in 4 hours by the use of 0.065 normal caustic soda.

Three plausible explanations were suggested for this rapid removal of sulfur from wool and the reduction of

the sulfur content to a value of about 1.85 per cent which remained constant during subsequent alkaline treatment:

1. The sulfur is largely concentrated in the exterior of the wool fibers.
2. Sulfur is present in wool in more than one combined form.
3. During the action of the alkali upon wool a portion of the sulfur is converted into a more stable combination in the wool protein.

Appropriate experiments with finely powdered wool eliminated the first explanation. That the second explanation is incorrect may be concluded from the results of other investigators who have shown that practically all of the sulfur in wool can be accounted for as cystine, and accurate analyses by Dr. Harris indicate that at least 96 per cent of the sulfur is in this form in the wool yarn used for these experiments.

The third explanation was established as follows: Ammoniacal sodium nitroprusside is a specific reagent for the sulfhydryl (—SH) group as in cysteine, the reduced form of cystine which contains the disulfide (—S—S—) linkage. After cystine has been treated with an alkaline reducing agent, e.g., potassium cyanide or sodium sulfide, it gives a positive test with sodium nitroprusside. Normal wool gives a negative test with this reagent, but after treatment with alkaline reducing agents this wool gives a positive test for the —SH groups. This result indicates that the alkaline reducing agents convert part of the cystine sulfur to the cysteine form. An alkali such as sodium hydroxide can accomplish this same change since the sulfur dissolved from the wool is converted to sodium sulfide by reaction with the sodium hydroxide, and this sodium sulfide can then function as an alkaline reducing agent. This conversation of cystine to cysteine would account for the non-removal of the residual sulfur since other studies have shown that cysteine is more resistant to the action of alkalies than cystine.

Previous treatment of wool with either oxidizing agents or reducing agents increases the rate of sulfur removal when the wool is treated with alkaline solutions. This is a matter of very practical importance, and it appears that the point of attack in the wool protein molecule, the weakest link in the chain, is the sulfur linkage.

Further information regarding the chemical relations and states of oxidation of sulfur in wool is required to round out our knowledge of the degradation of wool by chemical action. New methods of analysis must be developed. It is relatively simple to determine the total percentage of sulfur in wool, but no satisfactory methods are at present available for determining the distribution of this sulfur among the various possible states of combination in the protein. The sulfur which has been removed from wool is apparently present in the alkaline solution principally as sodium sulfide since it can be completely oxidized to sulfate by means of bromine and hydrochloric acid. This would not be possible if the cystine had merely been removed as such from the wool by the action of the alkali.

We realize with the utmost interest that the practical importance of the lability of sulfur in the chemical processing of wool, as brought out by this research, is paralleled by its similar importance in the fields of biochemistry and medicine. It is accepted by authorities in these fields that the oxidation-reduction balance of sulfur is one of the controlling factors in animal metabolism, and that it is related to the pathological conditions which result in such diseases as arthritis, diabetes and cancer. In this development of our research project, Dr. Harris and the committee have been in direct contact with a number of research workers in biochemistry and medicine. Further results relating to the chemical relations of sulfur in wool protein will be of direct interest and value to investigators in these fields, and mutual benefit will be derived from close cooperation.

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Proceedings of the American Association of Textile Chemists and Colorists

Color:

Its Characteristics and Phenomena*

By C. EARL HAYS

FROM a scientific standpoint the romance of color, as referred to in the shade of objects, is a most interesting study. There are many angles from which to consider the subject. The light to which a body is exposed, for example, naturally has much to do with the effect produced. Also, the composition of the matter composing the body, similarly affects the result.

As early as 1672, Sir Isaac Newton gave us a breakdown of the Spectrum in a broad way, followed by further research along this line by Wollaston, in 1802. Fraunhofer, in 1814, was the first to give us definite means for the division of the Solar Spectrum and assign a unit of measurement to the various points. Angstrom, who followed up the work of Fraunhofer, is generally credited with completing these measurements which today bear his name, viz; Angstrom units. Other noted physicists, such as Kirchhoff, Stokes, Stewart, and Rowland, have given us much valuable data in the study of color measurements.

We have learned from the experiments of these physicists that as white is composed of practically all color wave lengths, the sensation registered by our eye is caused by equal amounts of the various color wave lengths being reflected between two points, the incident light and the reflecting object. In the case of a color other than white, the unbalanced effect of the absorption of some of the waves would give the effect of that particular color represented by those reflected waves.

An object is said to be of a certain color when, upon subjecting the body to a white light, it absorbs all of the waves except that specific color wave effected through some method of pigmentation or other color matter processing. For example, should a piece of cloth, treated with a yellow dye, be exposed to a white light, it would absorb all other color waves except the yellow ones which would be reflected and in turn register that wave length sensation through our optical system (of which little is definitely known in respect to color). This above condition is also true of a color composed of more than one of the primary colors.

In considering the color of an object three things must be considered. There is one condition called "body absorption", as when a transparent substance which reflects color, gives the same color sensation when white light is viewed through it. Then we have what is known as "surface reflection", as from any object with a high reflection quality, such as a metal. However, with this last named condition we know that we can produce an entirely different result in color when the surface of the metal has been hammered thin and then we produce an effect of various colors called "fluorescence"; in other words, we have a combination of the two former conditions and produce the transmitted light of one effect while looking at the body, and from the side we produce the reflected effect.

We speak of the blue sky, blue or green water, or the white smoke. This condition is caused by the longer waves such as constitute the colors red and yellow passing by the small particles composing the element while these small particles pick up and reflect the shorter waves such as cause the colors green or blue.

Color also has weight, dimension and effect. It is greatly influenced by environment. For the purpose of illustration let us consider a heavy shade together with an equal amount of a very light shade. In a pattern the heavy shade would far outweigh the lighter one, and to form a balanced effect it would be necessary to have two or even three times as much of the pattern composed of the lighter shade, to offset the effect of the heavy one.

Colors, when properly balanced, blend and have beauty which will hold interest. This blending is done to produce a psychological effect. Various combinations are formed for the purpose of appeal or to definitely distinguish an article or garment.

The soft or warm shades cause the psychological effect of far-sightedness, while heavy shades are cool and appear to retire. In color strength a far greater variation can exist visually in heavy shades, while a very slight variation is noticeable in pastel shades. The variation in the darker shades might be, for example, 5% and it would be rather difficult for the human eye to observe while ½ of 1% in a pastel shade would be noticed immediately.

By careful selection, color can be used for many purposes. It can be used to create distinction or beauty, or even utility, and by careful contrasting of colors, shades,

* Presented at meeting, Northern New England Section, January 4, 1935.

and tone can be made to cause a pleasing sensation to the eye; this is accomplished when the frequency in reflection of one shade is made equal in reflection to that of the contrasting shade.

To further illustrate the point, let us form a chart in this manner by selecting the six points of the Spectrum and place them in a circle. (Fig. 1)

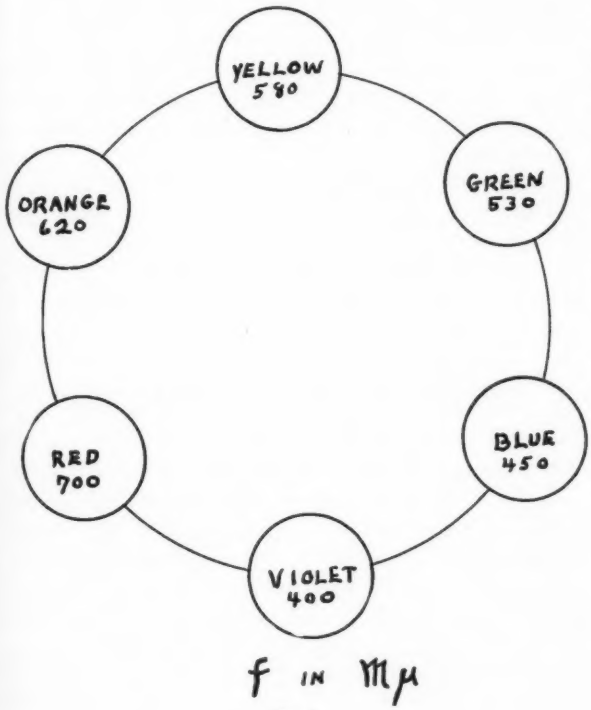

Fig. 1

Adjacents, such as yellow and orange, green and blue, form a soft pleasing effect when used together because the one is composed partially of the other. Opposites, such as green and red, yellow and violet tend to distinguish each other. In other words, adjacents will harmonize, while opposites will form a contrast.

An optical illusion is formed when colors are juxtaposed. They appear to blend automatically. For instance, if RED and BLUE are juxtaposed, using narrow lines, they would appear to mix and cause a darkened effect, this darkening is due to the two colors being opposites. If adjacent or related colors are used, in the same manner, the result would be brighter. Now to produce an entirely different result, select these same colors, RED and BLUE, and place them in the same manner, side by side; but in much broader lines they tend to isolate each other.

In order to get a comprehensive idea of the Visible Spectrum, it is well to know its position in the Etherial Spectrum and from this point let us take the Etherial Spectrum in all of its ramifications as we know it today. We find portions being used for radio telegraph. Next in order we have the Radio Broadcasting band, the short wave radio band, these three bands occupy approximately two-fifths of the Hertzian band and the remaining three-fifths are at present not being used. Next in order we have the infra-red or heat ray section, which is divided into three portions, the last portion includes the solar rays which we receive on earth. As this band is adjacent to the visible spectrum let me call your attention to those heat rays we sometimes see rising from a pavement in the summer season, when the sun is saturating the pavement with its rays, or to the rays we sometimes see under certain conditions rising from the steam radiators in our homes. In my opinion these vibrations have a definite color wave length, but they are not at the present time within a visible range. The next wave band is that small portion which is visible to the human eye and called the visible spectrum. This band ranges from the longer red rays to the violet or shorter rays.

In this visible range we find our primary, or unit colors, red, yellow and blue, from which other colors are basically produced and in addition to these primary colors we find the secondary colors, orange, green and violet. The frequency of the waves above the red are too long for our eyesight and those waves below the violet are too short.

Following the visible wave band is the ultra-violet, which extends into the X-rays, these are followed by the little known gamma bands and finally the last and least known cosmic ray bands. It is known however, that radium throws off gamma rays and we now know that cosmic rays emanate from interstellar space.

We find that the limits of our normal vision ranges roughly from 4,000 to 7,000 Angstrom units. If we were to lay out these measurements along the color spectrum with all its gradations it would be found that each variation of the primary colors had a definite wave length, or in other words, a specific number corresponding to the amount of light reflected from a definite light source.

As previously mentioned the quality of the true white light, as well as the composition of the matter, have much to do with the effect produced, but we did not consider the defects of human vision nor the fatigue of the human eye. These two factors are of the utmost importance, for even though we had the true white light and all other conditions were ideal, either a slight defect of vision or eye fatigue would cause us to lose the effect of the color.

In practically every other art, the artist has tools and gauges for every branch of his work, to be used in conjunction with his training and ability, but for the matching of colors the colorist has heretofore had to depend entirely on his eyes and many times under the most adverse conditions, but today, science has given us a tool which, when properly arranged together with other apparatus, can be

made to guide the man working with colors along the right path, no matter what the surrounding conditions happen to be. This tool is called the photo-electric cell, we sometimes hear of it spoken of as an "Electric Eye." This device is consistent in its opinion and is not influenced by any differences of opinion, nor subject to fatigue, such as is experienced by the colorist.

My previous work having been with light sensitive elements, I had seen the need of some device to aid the colorist, to act in the same capacity to the colorist as the micrometer to the skilled machinist. This led me to do some research work along this line. As a result, I built what many already know as the "Telecolor" (Fig. 2).

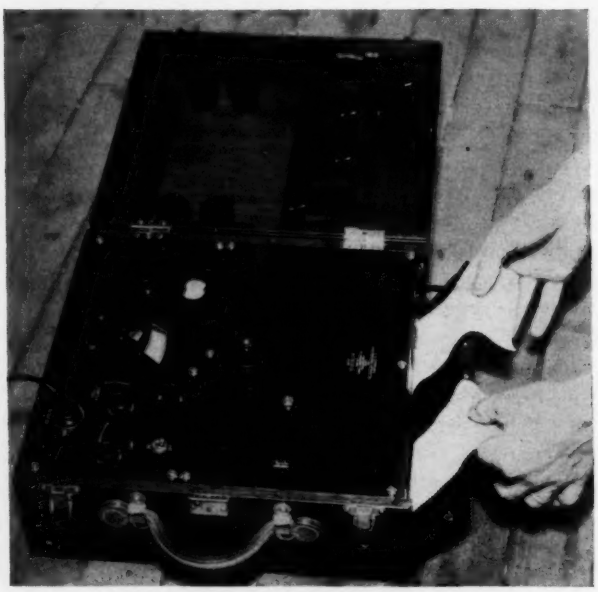

Fig. 2

This instrument incorporates photo-electric cells combined with other sensitive devices, for the purpose of distinguishing very slight differences of either color or tone of a specimen. The results are based on the value of reflected light as heretofore explained. It does not give a true spectrophotometric curve and its value lies in its use for comparative results. Heretofore, a constance source of energy has been most difficult to obtain, and this instrument does not depend on any external energy for its operation, with the exception of that used for the excitation of the light source. Also by introducing the necessary apparatus to amplify the reading of the changes or functions of photo cells, there is a danger of causing an accumulative error, which might tend to mislead one making a test. It was therefore felt that by an accurate but simple device, independent of extraneous changes, a colorist could be guided along the path of true matching with the slightest of tolerance error. It was further felt that an instrument for this purpose should be so designed that a chemist would not be expected to be familiar with mechanical devices or the method of making numerous electrical connections, and in my device this thought was kept in mind, with the result that the only change necessary for the satisfactory operation is the replacement of the light source, should it burn out or break from an unnecessary jar. Various types of photo cells and light sources were tested before the present types were accepted, and these photo cells have a long life expectancy.

As previously inferred, this instrument was not intended as a "cure all" for the color industry, but was meant as a "measuring stick" for color, and a guide for the colorist to follow.

At this point the speaker made some tests to familiarize the members with the operation of the instrument and answered questions concerning the uses of the instrument. After this the instrument was submitted to the group for their own operation at the conclusion of the meeting.

SPRING MEETING, SOUTHEASTERN SECTION

THE Spring Meeting of the Southeastern Section convened Saturday, April 27, 1935, at 6:30 P. M., at Auburn, Alabama. An inspection tour of the Textile School at A.P.I. was made by those arriving in the afternoon. An enjoyable dinner was served at the Thomas Hotel in the evening, at the conclusion of which L. N. Duncan, President, A.P.I., made an address of welcome to the members, and asked that they return whenever they desired.

J. W. Stallings, Manager, Charlotte Laboratory, Stein, Hall & Co., next delivered a paper on "Starches and Their Textile Application," following which an open discussion of this topic was led by L. L. Bamberger. The final paper was delivered by R. V. Irwin, International Salt Co., Charlotte, N. C., on "The Application of Salt Brine to Textile Processes," in which he gave the advantages of the Lixate process for using mined salt.

C. A. Jones, Chairman, then read a letter from William Kelly of the South Central Section in regard to the annual meeting to be held in Chattanooga in December, and appointed a committee to confer with the South Central Section concerning this.

C. B. Ordway, of A.P.I., was chosen to serve as Councilor for the Section for the ensuing year.

It was decided not to hold a summer meeting, but to hold the fall meeting in Atlanta, the date to be arranged later.

Respectfully submitted,

W. F. CRAYTON,
Secretary.

AMERICAN
DYESTUFF
REPORTER

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THE SUPREME COURT'S DECISION

"THIS decision of the court makes codes of fair competition unenforceable as a matter of law; and in deference to that ruling all methods of compulsory enforcement of the codes will be immediately suspended . . ." These words of Donald R. Richberg, chairman of the National Industrial Recovery Board, following the announcement of the decision of the Supreme Court in the Schechter test case, sounded the end of the NRA, at least as far as its present set-up is concerned. Just what the President intends to do now,—whether to frame a new law or let the NRA drop on its expiration date, June 16th,—remains to be seen and at the time of this writing there is no indication of his plans.

The effect of this ruling on the textile field is problematical at present but it is believed that there will be no drastic upheavals as most of the mill workers have their contracts with the mill owners. In the textile chemical field no effect is anticipated as this industry has been well governed by itself exercising such wisdom, common sense and restraint as had made no external regulation necessary. In many fields a sense of relief will be experienced with the lifting of outside regulation which, in many cases, was imposed by persons who had little or no knowledge concerning the business field over which they had control. According to the Institute of Dyers and Printers no immediate effect in this field is anticipated, depending to a certain extent upon whether a new bill is drawn to take the place of the outlawed act. It is stated that without some legislation there is the possibility of a gradual breakdown of the improvements which have been made in this industry.

The general consensus of opinion seems to be that the NRA had outlived its usefulness and that its end would be eventually forthcoming although the decision of the Court came to most as a surprise at this particular time. The decision of the Court merely voiced the opinion of the majority of people who had already decided that this statute was dead. It is gratifying that the decision of the Court was unanimous; now there can be no claim of a minority victory; it represented the viewpoint of both the liberal and conservative minds of the Court.

We can say no more at this time because of obvious readjustments that may take place and which will have effect on business.

● SURVEY OF WORLD CHEMICAL MARKETS

A world-wide survey of the CHEMICAL INDUSTRY completed since the beginning of 1935 has just been issued by the Commerce Department's Chemical Division, and is now ready for distribution, according to advices from C. C. Concannon who heads that branch of the Department's trade promotion activities.

This survey consists of a booklet of more than 100 pages which deals at length with chemical developments in 26 foreign countries and the United States during 1934. Foreign and domestic trade, production, new plant construction, modernization, plant extensions, cartels, trade agreements, new products, and all other factors necessary to round out a world picture of chemical developments during the year, are included in the study.

The whole field of chemicals and allied products, including heavy chemicals, chemical specialties, fertilizers, paint products, naval stores, medicinals, toilet requisites, dyes, plastics, coal-tar and non-coal products are covered in each country where they are of importance.

This survey, Trade Information Bulletin No. 823, can be obtained from the Superintendent of Documents, Government Printing Office, Washington, D. C., or from any of the Department's District Offices located in the principal centers of the country, at 10 cents per copy.

● SPECIAL COURSES FOR MILL MEN

The Textile School of North Carolina State College announces that during its six weeks summer session, which opens June 12, special courses will be given for mill men who wish to supplement their practical experience with some technical training. These courses will be taught by regular members of the Textile School faculty and will include work in various phases of Yarn Manufacture and Knitting, as well as elementary and advanced work in Designing and Fabric Analysis. The courses have been arranged so that those in attendance can devote all their time to the work in which they are especially interested.

During the same period the Department of Field Crops will conduct its annual summer course in Cotton Classing. This course will be taught by Dr. John B. Cotner, a licensed Government grader, who has successfully taught cotton classing courses at State College for many years.

Arrangements have been made so that mill men can divide their time between cotton classing and textile subjects, if they desire to do so.

Further information regarding these courses can be secured from Dean Thomas Nelson or Dr. John B. Cotner by writing them at State College Station, Raleigh, North Carolina.

In Memoriam

William C. Kaesche

Secretary

SANDOZ CHEMICAL WORKS, INC.

With a feeling of deepest sorrow we record the death on May 21st, 1935, of William C. Kaesche at his home, "The River House," 435 E. 52nd Street, New York City.

Although appearing to be in the best of health, Mr. Kaesche was suddenly stricken by a heart attack.

The loss of his lovable personality will leave a void in the lives of his many friends in the social and business world.

Born in New Rochelle, New York, July 31st, 1898, he attended Barnard and Lawrenceville, New Jersey, schools.

From 1917 he was employed under his father, Max B. Kaesche, President of F. Bredt & Company, continuing in this position when, in 1920, F. Bredt & Company was merged with Sandoz Chemical Works, Inc.

Mr. Kaesche was an ardent golfer, having at one time been golf champion of the Westchester Country Club. He was a member of the New York Athletic Club, the Winged Foot Club and the Apawamis Country Club.

Attributive no doubt to his love for athletics, Mr. Kaesche will be remembered for his active, vigorous character which earned him a host of loyal friends. He possessed an unusually alert mind and spent many hours perfecting a technique in bridge which was the envy of his friends and associates.

Funeral services were held at his home and interment was at Woodlawn Cemetery, New York City.

Mr. Kaesche is survived by his widow the former Miss Alice Louise Scharman, a son, William C. Kaesche, Jr., a brother, Max B. Kaesche, a prominent figure in the dyestuff business and widely known amateur golfer, his mother, Mrs. Max B. Kaesche and two sisters, Mrs. Harris B. Fenn of Ridgewood and Mrs. Herbert Willy Meyer of New York.

TECHNICAL NOTES FROM FOREIGN SOURCES

Delustering of Artificial Silk, with Reference to Damaging of Fiber

F. Ohl—Monats. f. Seide u. Kunst-Seide 38,342 (1933)—The previous part of this paper has already been commented upon in these columns, but appears, from the abrupt manner in which this section begins, to have been broken off from considerations of available space, rather than as a termination of a logical division. Exigencies of space, however, are peremptory.

(In reference to the matters dealt with in the closing part of the previous section) the specimens delustered with a barium salt and sulfuric acid appear to be the least of all resistant to storage for a considerable period, though in their preparation great care was taken to wash thoroughly. Even when sodium sulfate was used as the precipitant, the goods showed evidence of tendering some time afterward, though the effect was less pronounced than in the case of sulfuric acid as the precipitating agent. It would seem that in any case the damage is greater in those specimens in whose preparation a wetting-out agent was used; but the author considers this result to be due, not to any specific action of the wetting-out agent from the chemical side, but to its use favoring the deposition of greater amounts of salts (does he not mean, more exactly expressed, the resultant formation of a greater amount of barium sulfate within the fiber, with its possible precipitation in a microcrystalline form, possessing microscopic but none-the-less real cutting edges?) He concludes that the best matte effects produced by barium sulfate involve also the greater danger to the stability of the fiber, and that the finishers, etc., who are to work upon such a fiber, must take care to leran how long and under what conditions the material has been stored, and how far tendering has already set in. The results do not show any difference in favor of either barium chloride or sulfuric acid (or sulfates) as the first or second bath.

The author has extended his studies to the delustering of viscose by means of barium chloride and sodium stannate, using the same general method as before. Fiber unbleached, and bleached with activine, was used. The goods were impregnated first in a bath containing 2 g. of sodium stannate (containing 43-44 per cent of stannic oxide) per liter, at 40° C., for 20 minutes, being worked meanwhile; then, without rinsing, immersed in a bath of barium chloride of 0.2 per cent at the same temperature, for 30 minutes. The preparations were made with and without the addition of a wetting-out agent (Praestabitoel V). The results could easily be divided into two groups, the matte effect being full and excellent in the case of the bleached fiber, the loss of luster in the unbleached material being less pronounced. Tests showed the permanence of the delustering to be excellent. The various lots were stored for different lengths of time, and tested, as before, for possible damage (longest period of storage, nine months). The table (No. 2) gives the results numerically, as in the previous part of the paper.

This sodium stannate method gave an actually greater breaking strength to the fiber. Some evidence of loss of this quality, on storage, was gained, but the loss was very slight compared to the rather serious figures obtained for the product of the sulfate method. Again, those specimens in whose preparation a wetting-out agent was used, showed less resistance to the effects of storage than did theh others. The author adds, that one must consider whether, at some period later than the maximum selected (nine months), deterioration might not set in suddenly (though he raises the point only as a question to be settled merely that the question may be permanently answered). Work was further carried out upon the third principal method of delustering commonly practiced industrially—the one involving salts of titanium. The method here used consisted in impregnating the goods with a titanium sulfate solution, 4 g. per liter, at 20° C., and warming during 30 minutes up to 85° C., finally centrifuging and drying. Modifications of the method consisted in autoclaving for 20 minutes at 2.5 atmospheres, and the use or omission of wetting-out agents. The various samples so obtained were examined as before. No increase in tensile strength was noted. Some loss in tensile strength was observed from storage, though in no case so serious as with the barium chloride-sulfate method. The autoclaved samples suffered most. The extent of delustering and permanence of effect were about the same as with the barium chloride-sulfate method, i. e., bleached goods were more delustered than the unbleached, and permanence of effect was excellent in each case.

The author also carried out work, according to these three methods, upon weaves, and the results were accordant with the above. Comment is made upon dyeing difficulties with the delustered fiber; dyeing has little effect upon the matte finish, least of all upon the sodium stannate and titanium sulfate products.

Ester-Salts of Leuco-vat Dyestuffs

German Patent No. 474,036 (25/III/'29) (*additional to G. P. No.* 424,981 *of* 27/X/'25)—*Durand-Huguenin A.-G.*—In the original patent, chlorsulfonic acid was employed to esterify the leuco-vat dyestuffs. The present addition specifies the use of a related compound instead, pyrosulphuryl chloride, Cl—SO_2—O—SO_2—Cl, which is cheaply prepared, and has the advantage over chlorsulfonic acid that a reasonably small amount of moisture present in the leuco-compound (which is difficult to get out entirely) does no harm, merely converting its equivalent of the pyrosulfuryl into chlorsulfonic acid (and is itself destroyed), which then also will obviously be available for forming the ester as in the original process.

Two examples are given.

Vat Dyestuffs (Benzanthrone Series)

German Patent No. 475,342 (27/IV/'29)—*I. G. Farben-Ind. A.-G.*—Reference is made to a certain amino-benzanthrone, produced by reduction of a nitro-benzanthrone, described in example 2 of addition No. 6435 to French Patent No. 349,531. The product, the amino-benzanthrone, is here fused with caustic alkali (in one case given, equal parts of caustic potash and ethyl alcohol are heated up to 150° C. as the alcohol distills off, and the amino-benzanthrone mentioned, m.p. 298° C., stirred in). After heating at 180° C., as long as dyestuff formation goes on, the melt is dissolved in water, heated to boiling, and aerated until the dyestuff is completely precipitated.

Two examples are given, the products dyeing cotton, one a strong green from a greenish-blue vat, the other gray from a blue vat.

Tetrakis-azo Dyestuffs

German Patent No. 477,914 (10/VI/'29)—*I. G. Farben-Ind. A.-G.*—A rather comprehensive, but clearly described procedure.

a—Amino-disazo dyestuffs are first prepared, of the type $A-N=N-B-N=N-C-NH_2$, in which A is any desired amine, B a component of the naphathalene series containing an amino-group and a position to which A can be coupled when diazotized, C a derivative of alpha-naphthylamine with its 4-position free for the AB mono-azo dyestuff to couple into. This last component, C, may contain sulfonic groups, and must contain substituted or free hydroxyl groups.

b—These disazo dyesuffs are diazotized and coupled to derivatives of J-acid, which may be substituted in the 1-position, and which must in any case carry heteronuclear side-chains which contain free and diazotizable amino-groups.

c—These tris-azo dyestuffs are diazotized and condensed with methyl-ketol or its dreivatives, or with pyrazolones or their derivatives, or with beta-ketonaldehydes, or with 1-3-dihydroxy-quinoline, or with sulfazones, or with salicylic acid or any of its derivatives which can act as second components.

The products all appear to dye cotton in clear green tones of good fastness to light, from neutral or alkaline baths. One would suspect that the J-acid-heteronuclear complex might be one which would tend toward producing green products, just as pyrazolones tend toward the yellow-orange group unless considerably modified by substituting groups with an opposing tendency.

Three examples are given.

● GENERAL RELEASES

General Dyestuff Corp. announces release of bulletins on the following products. Copies are available upon request.

Rapidogen Orange R—a reddish orange dye said to be of very good fastness to light, washing and chlorine and to retain its shade in continued soaping.

Igepon T—an auxiliary for processing vegetable and artificial fibers. This 18 page booklet describes the properties of Igepon T, its application, and a variety of uses. Among the uses described are: kier boiling; bleaching; dyeing cotton, rayon, mixed materials, etc.; sizing, desizing and finishing; and cleaning of dyeing machinery. There are also numerous illustrations.

● BUTTERWORTH BULLETINS

H. W. Butterworth & Sons Co. have available three new bulletins on the Tube-Tex machine. One deals with the single pass extracting and processing machine which replaces centrifugal extracting and which is available as a separate unit or as part of any Tube-Tex range. Another describes the three Tube-Tex combinations: "A" for wet spreading, squeeze roll extracting, drying, control conditioning and folding; "B" for wet spreading, squeeze roll extracting or processing (if desired), drying, control conditioning and folding, and "C" for wet spreading, squeeze roll extracting and processing (if desired), drying, control conditioning and folding. The third circular is devoted to the Tube-Tex high speed steamer and finisher. Copies of any or all of these bulletins can be obtained on request.

● GEIGY DYE

Geigy Company, Inc., has released a color card on the following dyestuff. Copies are available upon request.

Wool Fast Navy Blue FTLN—a product similar to the FTL brand having same general properties but with improved fastness to light. It is a weak acid wool dye and it is stated that it should find a ready use in the production of bright fast navy blues on wool piece goods for both men's and ladies' wear. It is further stated that this dye should be of special interest for the dyeing of hosiery. It is said to be not affected by chrome and therefore useful for shading Eriochrome colors. Viscose and cotton are slightly stained, which, it is said, gives a pleasing bright blue to checking threads.

● 1935 FALL WOOLEN CARD

The regular edition of the 1935 Fall Woolen Card, portraying forty-eight colors in woolen fabric, was recently released to members of the Textile Color Card Association.

In addition to important basic shades, the card contains two featured color groups designated as "Rustiques" and "Touchdown and Ski Colors." The first-named collection consists of hazy pastels and rich Autumn tones especially suitable for dresses and children's wear. The lively "Touchdown and Ski Colors" have special merchandising appeal for the football and Winter sports seasons.

Because of the continued fashion favor for color combinations, the shades are especially grouped in the card to suggest new harmonies and contrasts.

OPEN FORUM

Rules under which this department of the REPORTER is conducted are as follows:

1. Anyone who has a technical problem on which he would like advice may submit it to the Reporter and it will be published in the first available issue.

2. Any reader who feels that he has a solution of the problem may forward his reply to us and if we feel that the reply is suitable it will be published in a succeeding issue.

3. For each such reply published the Reporter will pay a minimum of $2.

4. In case there is more than one answer to the same question, the one covering the subject in the most complete and authentic manner will be published, or if two answers are sufficiently different both will be published. If two answers are considered equally good, the one first received will be given priority.

5. The Reporter will assume no responsibility whatsoever for the accuracy of the answers submitted—it is simply offering a vehicle for the public expression of its readers' opinions.

6. Both the questioner and answerer must send in his complete name and address. When requested we will publish only the initials or other identifying insignia.

Question

26—I am interested in obtaining information concerning the composition of finishing compounds as applied to silk and rayon piece goods. Also methods for the laboratory analysis of such finishing compounds.—**S. A.**

Answer:—There are a great many substances used in sizing silk and rayon piece goods. The chief filling materials aside from dullers such as titanium oxide, calcium carbonate, etc., are carob tree gum, gum tragacanth, gum arabic, agar agar, Irish moss, alginates, glue, gelatin, and starches. A number of synthetic gums are also in use. Softening agents consist of soaps, sulfonated oils, fatty alcohols, hydrogenated oils, mineral oils, and similar substances. Glycerin is frequently used for hygroscopic purposes.

Hygroscopic agents and oils are used very little on

TABLE 1

50 GRAM SAMPLE OF PROPER CONSISTENCY IS TREATED AS FOLLOWS

A.—Test with litmus and neutralize if necessary.

B.—Add slowly with constant stirring 3 c.c. of 10% ammonium chloride. Then add 10 volumes of absolute alcohol. Shake well. Allow to stand 24 hours. Filter on suction filter and suck almost dry.

Filtrate from B contains any soaps, oil, fats, glycerine, sugar, etc.	Precipitate from B contains inorganic salts, glues, gums, starches, etc.
	E.—Shake twice in a flask using 50 c.c. of cold water each time. Filter.
Distill off the alcohol. Transfer residue to a platinum dish. Evaporate to a syrup and finally dry in an oven at 105°C.	F.—The residue from E is dissolved by pouring 100 c.c. of hot water through the filter several times.
Fats and oils are indicated by precipitating. Extract the dry mass by dissolving in water and shaking up with ether in a separatory funnel. The oil is determined by the ordinary methods of oil analysis.	G.—The filtrate from F is precipitated with basic lead acetate solution. Filter.
C.—The water layer is acidified with hydrochloric acid and boiled for 5 minutes. Oily layer indicates soap or sulfonated oil. Extract with ether as before. Test the water layer for sulfates to confirm the presence of sulfonated oils.	The filtrate from G is divided into two portions—H and I.
D.—Evaporate the water layer from C to a syrup. Cool and add 5 volumes of ether alcohol 1-1. Allow to set 24 hours in a stoppered flask and filter.	J.—The residue from G contains any arabic, carob tree, tragacanth and other gums present. Fifty per cent acetic acid is poured over the mass and let stand several hours before filtering.
The precipitate from D if any is dissolved in water. Made alkaline with caustic soda and tested for glucose with Fehling's solution. Acidify with sulfuric acid. Boil 10 minutes. Neutralize again with caustic soda. Further precipitation with Fehling's solution shows presence of sucrose.	Test filtrate for glycerin by making regulation Schiff's Reagent-aerolein test, i.e., absorb in potassium bisulfate, etc.

The combined filtrates from E are divided. The usual qualitative tests for inorganic salts are made on one portion. The other portion is tested for dextrin by the simple iodine test.	The residue from F is albumen. This is verified by testing with Millon's Reagent.	Neutralize H and test with iodine for starch and dextrin.	Test I with tannin solution for glue and gelatin. Confirm by dilute hydrochloric acid. Glue and gelatin tannin precipitates are partly soluble. Starch tannins are not soluble.	The filtrate from J is treated with Fehling's solution and caustic soda until the lead sulfate redissolves and allowed to stand. Carob tree gum will precipitate if present.	Dissolve the residue from J in hot water. Filter to remove insoluble lead salts. Add an excess of 5% tannin soution. Turbidity shows Irish moss. Add hydrochloric acid. Increased turbidity shows Agar Agar. Clear solution shows tragacanth and similar gums. It is almost impossible to distinguish definitely different gums of the same class by any ordinary test.

Fehling's solution—See any textbook or quantitative analysis.

Millon's Reagent—See any quantitative textbook.

Tannin solution—5 grains of tannic acid are dissolved in 100 c.c. of distilled water.

Schiff's Reagent Aerolein test—See textbook for Schiff's Reagent. Add enough bisulfate to make a dry powder. Pour into dry test tube provided with a one-hole stopper and a bent glass tube. On heating the mixture aerolein will be found if glycerine is present. This will restore the red color to Schiff's Reagent.

TABLE 2
REACTION OF TYPICAL SIZE BUILDERS

	Alcohol	*Fehling's Solution*	*Basic Lead Acetate*	*Caustic Soda & Copper Sulfate*	*Iodine Solution*	*Tannin Solution*
Gum Arabic	white flocculent precipitate	blue gelatinous mass which does not change on heating	white gelatinous precipitate	blue gelatinous precipitate	no action	no action
Algaes and Irish Moss	white ppt. in strong alcohol	no action	white precipitate	blue flocculent precipitate	slight violet color	solution gets turbid
Carob Tree Gum	white flocculent precipitate	blue gelatinous mass which does not change on heating	opaque white precipitate	blue gelatinous precipitate	no action	brownish gelatinous precipitate which redissolves on heating
Glue and Gelatin	white ppt. in strong alcohol	violet color	turbidity in strong solutions	violet color	no action	precipitates as a lump partly soluble in dilute hydrochloric acid
Gum Tragacanth	white ppt. in strong alcohol	no action	opaque white ppt.	blue flocculent ppt.	no action	no action
Albumen Solution	coagulates	violet color	opaque white ppt.	violet color	no action	precipitates
Starch	white ppt. in strong alcohol	no action	turbidity	blue ppt.	blue or violet color	precipitates in strong solutions
Dextrin	pasty precipitate	no action cold—red precipitate on heating	no action	no action cold—red ppt. on heating	brown, purple or red color	slight turbidity in strong solutions

silks. Fillers are used to a great extent. The reverse holds true for rayons.

There are no positive tests for mixtures of gums in the same class which will readily distinguish them. The scheme shown here (Tables 1 and 2) for analysis may be helpful after a little practice. The presence of fatty alcohols in a size mixture is discussed on page 137 of the March 25th Dyestuff Reporter.—B. H.

● SILK SOAKING OILS

The Berkeley Products Company announces that the need of a silk soaking oil that will successfully perform all necessary functions and, at the same time, will not produce corrosion of needles, etc., has caused the Berkeley Products Company to obtain from the Emery Industries, Inc., the full right to market in the soaking field, oils based upon the Twitchell patents, and utilizing the principle of sulfonated mineral oil as a dispersing agent for other non-corrosive oils.

It is also announced that Mr. Carvin, president of the company, has arranged his other business interests in such a way that, starting immediately, he can devote the bulk of his time to the marketing of these new products. Mr. Carvin is well known in the throwing industry, having been actively engaged in that industry for the past ten years. The preliminary work that led to the development of these oils was carried out in the Conshohocken Plant of the Clifton Yarn Mills by Mr. Carvin and Mr. Hathorne, while Mr. Carvin was actively engaged in directing the activities of that plant.

CLASSIFIED

The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i.e., help wanted, machinery or supplies for sale—the rate is $5.00 per column inch or less per insertion.

WANTED: A chemist thoroughly familiar with textile processes and the manufacture of chemical specialties for the textile trade. Must be thoroughly acquainted with the application of such chemicals on a practical scale. Write Box 860, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.